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**Weapons Systems Division**
**Aeronautical and Maritime Research Laboratory**

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## ABSTRACT

Melt-castable NTO/TNT explosive formulations have been identified as possible candidates to replace RDX/TNT to meet Australian Insensitive Munitions obligations. Such formulations could be processed in existing ADI industrial facilities with minimal alterations to plant.

A baseline 50:50 NTO/TNT formulation, designated ARX-4002, has been developed. ARX-4002 can be processed using traditional melt-cast techniques and exhibits reduced sensitiveness in comparison to Composition B and H-6. The molten slurry is less prone to sedimentation problems, however, viscosities are increased and lower solid loadings possible than for Grade A RDX.

Velocity of detonation and detonation pressure were experimentally determined at 7370 m/s and 22.6 GPa respectively. Performance of ARX-4002 is thus less than for Composition B, however, the intrinsic sensitivity of ARX-4002 is expected to be greatly reduced as indicated by the large critical diameter (22.5-25.5 mm).

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## Executive Summary

The drive to achieve Insensitive Munitions (IM) compliance in modern weapons systems has mainly centred on the development of polymer bonded explosives (PBXs). Australia has a limited ability to formulate and process PBXs and has invested heavily in recent years in modern melt-cast manufacturing and processing plants at ADI. An IM approach that could utilise existing plant is thus desirable.

Melt-castable NTO/TNT compositions have been identified as possible candidates to meet IM criteria and could be processed on Australian plant. A baseline 50:50 NTO/TNT formulation (ARX-4002) has been developed which can be processed using traditional melt-cast techniques. The composition is less prone to sedimentation problems than RDX/TNT and shows a reduced sensitiveness to hazardous stimuli. Slurry viscosities, however, are increased and lower solid loadings achievable than for Grade A RDX.

Velocity of detonation and detonation pressure were experimentally determined at 7370 m/s and 22.6 GPa respectively. Performance of ARX-4002 is thus less than for Composition B (60:40 RDX/TNT), however, the intrinsic sensitivity of ARX-4002 is expected to be greatly reduced as indicated by the large increase in critical diameter (22.5-25.5 mm, *ca.* 3-5 mm for Composition B).

The study has allowed the necessary experimental techniques to be developed to handle more complex formulations with improved explosive and IM properties.

# Authors

**Matthew D. Cliff**
Weapons Systems Division

*Matthew Cliff completed his Honours degree at Deakin University in 1991 and his PhD in organic chemistry at the University of Wollongong in 1995. He commenced work at AMRL in 1996 and has worked on a range of tasks looking at new nitration methods, synthesis of energetic materials and PBX formulation and evaluation. In 1998/1999 he was attached to the Defence Evaluation and Research Agency, Fort Halstead in the UK and is currently looking into melt-castable Insensitive Munition fills and reactive metals for use in explosive formulations.*

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**Matthew W. Smith**
Weapons Systems Division

*Matthew Smith completed his Honours degree at the University of Adelaide in 1996 in organic chemisrty. Following several months looking at organometallic compounds, he commenced work at AMRL in 1997. He has worked on a number of tasks examining energetic materials and is currently carrying out research into new insensitive formulations.*

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# Contents

# 1. Introduction

The past decade has seen an increased emphasis on meeting Insensitive Munitions (IM) criteria for new and upgraded weapons systems in the Western world. Australia is not immune to these trends and has an active research program to develop and assess intrinsically less sensitive explosives. Research into and implementation of IM in Australia is supported by a Defence IM policy, DI(G) LOG 07-10, which states 'IM are to be introduced into Service with the Australian Defence Organisation where it is sensible, practicable and cost-effective to do so' [1].

The major research thrust worldwide to achieve IM compliance has been the continued development at PBX's, employing either inert or energetic rubbery binder systems, to confer insensitivity into an explosive warhead. While this has met with considerable success, such systems can be unsuitable for high acceleration articles such as field and naval artillery shells, or are expensive and time consuming to process. Australia has a limited capability to formulate and process PBX's for the ADF. Mixing facilities at ADI are limited to a single 30-gallon mixer for Nulka rocket motor and Penguin warhead (PBXN-109) production. Additionally, considerable capital has been invested during the 1990's on the relocation and modernisation of the RDX/TNT manufacturing and processing plants at ADI Mulwala and Benalla. An IM approach that would enable existing plant to be utilised is thus desirable for Australia.

NTO (3-nitro-1,2,4-triazol-5-one) has been identified as a potential RDX replacement in Composition B type formulations [2]. NTO/TNT based melt-cast explosives have shown promise in the USAF insensitive General Purpose bombfill AFX-645 [3] and for use in extended range artillery rounds developed in South Africa (NTO within an HNS grain-modified TNT matrix) [4, 5]. The benefits of such formulation types for Australia include;

1. an existing broad knowledge base of TNT-filled ordnance at a scientific and industrial level,
2. existing industrial plant,
3. well characterised performance and long shelf-live,
4. low cost,
5. ease of demilitarisation.

It must be recognised, however, that problems of cracking inherent to TNT-based fills may still create difficulties.

The aim of this study was to investigate a baseline NTO/TNT melt-castable formulation, develop processing techniques and determine the hazards response and performance of a simple composition prior to the assessment of more complex formulations.

# 2. Rheological Studies

A 50:50 NTO/TNT formulation, designated ARX-4002 [6], using a single medium NTO particle size distribution (200-400 μm) was used for this study. NTO was obtained from Dyno Defence in Norway as water recrystallised particles and used directly as received. SEM micrographs of the NTO grade used are shown in Figures 1 and 2.

## 2.1 Formulation

Two formulation methods were used for processing ARX-4002. Normal melt-casting techniques could be used for both methods (open casting into moulds or biscuit-filling for charges of large diameter).

**Method 1**
Dry NTO was added incrementally to molten TNT at 90-100°C and hand stirred until thorough wetting of the solids was achieved. The resulting slurry was considerably more viscous than for an identical solids loading of Grade A RDX [7]. A maximum solid loading of approximately 53% was possible using a monomodal medium grade NTO, with greater loadings unable to be processed.

Method 2, using a vacuum mixing process, was found to produce molten slurries of reduced viscosity and solid castings free from voids, and was the formulation technique of choice.

**Method 2**
NTO was incrementally added to TNT as in Method 1 above and the slurry mixed for 10 minutes under a low vacuum (15-20 mmHg) to remove occluded air from the mix. The resulting slurry showed a dramatic reduction in viscosity. This method of processing was able to sustain monomodal NTO loadings of close to 55% whilst maintaining processability.

Mechanical polishing of ARX-4002 samples processed by the above methods was achieved using a modified Composition B polishing technique [8] and is detailed in Appendix A. SEM analysis of polished sections of ARX-4002 produced by formulation methods 1 (Figures 3 and 4) and 2 (Figure 5 and 6) showed an even distribution of NTO throughout the TNT matrix. NTO particles appear to be well bound within the matrix and have been thoroughly wetted whilst in the molten phase. Numerous voids were evident within samples formulated at atmospheric pressure, most probably due to occluded air in the slurry. These were not evident in samples formulated *via* Method 2, which gave castings containing few detectable voids. Vacuum mixing is thus necessary to obtain high quality charges of ARX-4002 and is the processing method of choice. Chloroform etched samples (Figures 7 and 8) clearly show the TNT grain structure, however, considerable debris was deposited onto the etched surface. This is most probably due to precipitation of dissolved material upon removal from the etching media and should be minimised by use of less volatile bromoform. (Bromoform was unavailable during this section of work and chloroform was used as a substitute).

## 2.2 Solids Sedimentation

NTO showed a lower tendency to sediment from molten TNT than equivalent loadings of Grade A RDX. ARX-4002 and 50:50 RDX/TNT were formulated by Method 1 above, open-cast into 25 mm diameter cylindrical moulds and allowed to solidify. The castings were sectioned into 10 mm high disks and the TNT dissolved off with benzene (NTO and RDX solubility in benzene was 0.013 and 0.05 g/100 ml respectively at 20°C). The percentage composition of each disk for the two castings is shown graphically in Figures 9 and 10. The composition of ARX-4002 remained fairly consistent throughout the charge length and showed no gross levels of sedimentation. The reference RDX/TNT casting, however, had a greater tendency for RDX settling, with more than 55% nitramine found in the bottom two disks and heavy depletion at the top of the charge. The reference composition was also more prone to coring at the top of the TNT-rich casting as a result of greater levels of shrinkage (Figure 11).

The low rate of NTO sedimentation seen in ARX-4002 is linked to the high viscosity of the molten formulation. Viscosity levels are high enough to retard sedimentation during the solidification process, however, this in turn prevents high solid loadings from being achieved. For Grade A RDX, particle morphology has been optimised to enable high loadings to be achieved. Thus at relatively low loadings (50%) the viscosity of the slurry is significantly reduced and results in an increased rate of sedimentation.

Formulation of ARX-4002 under vacuum gives reduced slurry viscosities and while an increased NTO loading is achievable, the rate of sedimentation at the 50% level would most likely increase. Time restrictions did not allow for the analysis of ARX-4002 castings formulated *via* Method 2.

# 3. Hazard Assessment and Vacuum Stability

Rotter Impact (F of I), Electrostatic Discharge (ESD) and Temperature of Ignition (T of I) testing was carried out according to the UK Sensitiveness Collaboration Committee Manual of Tests [9]. BAM friction testing was performed according the UN test manual for the transport of dangerous goods [10], and vacuum stability tests run for 48 hours at 120°C. A summary of sensitiveness and vacuum stability data is given in Table 1. Evolved Rotter Impact gas volumes are given in parentheses.

*Table 1. Sensitiveness and Vacuum Stability Data for ARX-4002 and other TNT-Based Formulations.*

| | ARX-4002 | Composition B* | H-6* | NTO (200-400 μm) | RDX (Grade A) |
|---|---|---|---|---|---|
| **F of I (ml)** | 180 (1.7) | 130 (4.2) | 170 (2.9) | 120 (4.9) | 80 (11.9) |
| **BAM (N)** | 192 | 112 | 112 | > 360 | 128 |
| **ESD† (J)** | > 4.5 | | | > 4.5 | 0.45 |
| **T of I (°C)** | 251 | 212 | 202 | 265 | 223 |
| **Vac Stab (ml/g)** | 0.10 | 0.15 | 0.39 | 0.10 | 0.02‡ |

* Grade A. † Lowest energy level (4.5, 0.45, 0.045 J) at which an event occurs. ‡ 120°C/48hrs.

Rotter impact testing of ARX-4002 showed the composition to be less sensitive to ignition by impact (F of I = 190) than the TNT-based Composition B and H-6. Similarly, BAM friction testing required considerably greater loadings to initiate an event. A loading of 192 N was, however, markedly less than for the neat NTO grade (200-400 μm) used in the formulation (> 360 N) and may have been partially due to the sample preparation. Solid TNT-based samples submitted for BAM friction testing are ground into a powder and sieved to remove particles greater than 500 μm. The test is then conducted on the screened material. Comparison of the friction results of ARX-4002 (192 N) to that of a finer grade of NTO (5-10 μm, 180 N) produced by air-jet milling of the coarse material showed a similar sensitiveness. This suggests that the process of sample preparation for ARX-4002 in which the NTO is finely ground possibly induce additional friction sensitiveness onto the sample, leading to a result similar to that seen for finer NTO grades. Further testing of other NTO grades and formulations is required to verify this result.

Both NTO and ARX-4002 were seen to have low sensitiveness to electrostatic discharge, with no ignitions evident at the maximum energy level used. The Temperature of Ignition was considerably higher than for Composition B and H-6. The T of I for TNT was seen to be 299°C, indicating that the rapid decomposition of NTO (T of I = 265°C for a neat sample) is responsible for the event onset temperature (251°C) observed for ARX-4002. Similar behaviour is seen for Composition B and H-6, in which RDX decomposition initiates events at 212 and 203°C respectively.

# 4. Thermal Assessment

The DSC and TGA traces of ARX-4002 are shown in Figures 12 and 13 respectively. Thermal analyses were performed in duplicate and the average onset temperatures and TGA weight losses presented in Table 2.

*Table 2. Summary of DSC/TGA Thermal Data of ARX-4002, NTO and TNT.*

| Material | DSC Onset (°C) | | TGA Weight Loss |
|---|---|---|---|
| | Endo | Exo | (%) |
| **ARX-4002** | 80 | 261 | 78.6 |
| **NTO** | - | 275 | 34.1 |
| **TNT** | 78 | 315 | 87.2 |

A reduction in decomposition onset of approximately 15°C is seen for ARX-4002 over the neat initiating energetic, NTO. This trend matches that observed in the T of I (Table 1). Event temperatures are approximately 10°C higher in the DSC and TGA runs in comparison to T of I experiments. This is possibly due to the inert atmosphere used in the DSC/TGA experiments preventing reaction with oxygen, hence decomposition is due to thermal stimuli alone. (For T of I experiments, the sample tube is open to the atmosphere and the contents able to be oxidised and initiate an event).

The exothermic decomposition onset of ARX-4002 represent a significant improvement over RDX and Composition B which thermally decompose in the region of 220°C.

# 5. Explosive Performance

## 5.1 Velocity of Detonation

Velocity of Detonation (VoD) measurement were performed in quadruplicate on cylindrical charges (41 mm dia x 200 mm length) open-cast to a density of 1.71 g/cm$^3$ (96.5% TMD). Charges were boosted with 50:50 pentolite 41 mm right cylindrical pellets and initiated with Resi 501 EBW detonators. VoD was measured *via* streak photography.

An average VoD for ARX-4002 from the four charges was 7370 m/s (Std Dev 25 m/s). This compares well with that obtained by other researchers (7340 m/s at $\rho$ = 1.74 g/cm$^3$) [11].

## 5.2 Chapman - Jouguet Pressure

Detonation pressure ($P_{CJ}$) was estimated from dent tests and compared to open-cast TNT [12]. Six cylindrical charges of ARX-4002 and six of TNT (41 mm dia x 200 mm length) were fired into stacks of mild steel witness plates to give average dent depths of 7.236 and 6.902 mm respectively. The $P_{CJ}$ for ARX-4002 was calculated at 22.6 GPa.

This figure closely matches that obtained from the relationship,

$$P_{CJ} = \frac{\rho D^2}{\gamma + 1}$$

where ρ = density ($g/cm^3$).
D = limiting velocity of detonation (km/s).
γ = ratio of the specific heats of the detonation product gases.

derived by Fichett and Davis from simple one-dimensional detonation theory [13]. Allowing γ to be equal to 3 (a good approximation for most high explosives close to TMD), then $P_{CJ}$ equates to 23.2 GPa, a difference of 0.6 GPa to that obtained *via* the dent test method. (Literature value = 23.1 GPa [11]).

## 5.3 Critical Diameter

The critical diameter ($d_{crit}$) of ARX-4002 was estimated to a first approximation using a stepped cylindrical charge of decreasing charge diameter. ARX-4002 was open-cast into a 25.5 mm diameter cylindrical charge and machined into sections of decreasing diameter. Section diameters were 25.5, 20.0, 17.5, 15.0, 12.5 and 10.0 mm, with each section being 40 mm in length (Figure 14). The charge was initiated from the 25.5 mm end and the detonation followed by streak photography until failure occurred. All three stepped charges failed to sustain the detonation within the 3rd section, indicating a critical diameter of $17.5 < d_{crit} < 20.0$ mm. Unconsumed sections of the charge below this point were recovered from the chamber floor.

In order to obtain a more accurate estimate of $d_{crit}$ a number of ARX-4002 cylindrical rate sticks with diameters in the range 17.0-20.0 mm were fired. Charges of diameter 20.0 mm, however, failed to sustain the detonation and indicated that the reaction had been significantly overdriven during stepped charge firings to give an underestimation of $d_{crit}$. A second series of rate sticks of diameter 25.5 and 22.5 mm were fired and the detonation again followed by streak photography. Sticks of diameter 25.5 mm were readily initiated from 25.5 mm diameter pentolite boosters, with the detonation front propagating through the charge at 7170 m/s (Figure 15). Charges of diameter 22.5 mm, however, failed to sustain the detonation (Figure 16). No residual explosive was found in the chamber after firing and was presumably consumed in low order events.

The unconfined critical diameter of ARX-4002 was determined to be $22.5 < d_{crit} < 25.5$ mm.

## 5.4 Detonator Sensitivity

Six small cylindrical charges (41 mm dia x 73 mm length) were assessed for their ability to initiate from Resi 501 and SSGT EBW detonators. As expected, all charges were unable to be initiated and a fractured cylinder was recovered from the firing point with no evidence of charring or ignition.

A summary of the explosive performance of ARX-4002 and Composition B [14] is given in Table 3.

*Table 3. Comparison of ARX-4002 and Composition B Explosive Performance.*

| Performance Parameter | ARX-4002 | Composition B |
|---|---|---|
| **Velocity of Detonation (m/s)** | 7370 | 7890 |
| **Detonation Pressure (GPa)** | 22.6 | 28.7 |
| **Density (g/cm$^3$)** | 1.71 | 1.72 |
| **Critical Diameter* (mm)** | $22.5 < d_{crit} < 25.5$ | 3.73, 4.24 |

* Unconfined.

The performance of ARX-4002 is considerably less than Composition B, with VoD and $P_{CJ}$ down by approximately 7 and 21% respectively. Greater NTO loadings would be required to improve the performance to a level comparable to the Composition B, the explosive fill used in the majority of ADF ordnance. The critical diameter, however, is considerably greater than that of Composition B and indicates that the intrinsic sensitivity of ARX-4002 would be reduced.

# 6. Discussion

Despite the performance of ARX-4002 being somewhat low, it has been shown that intrinsically less sensitive NTO/TNT melt-cast compositions can be formulated and processed with relative ease in a manner similar to RDX/TNT. The major difference between the composition types is the increased viscosity and accompanying low solid loadings achievable using this particular NTO grade. These difficulties, however, should be overcome by tailoring of the NTO crystal habit. Researchers at RMC Shrivenham using *N*-methylpyrolidinone recrystallised NTO have reported successful incorporation of 60-70% NTO within a TNT matrix [15]. Utilising this NTO type or bi and trimodal NTO blends along with the vacuum mixing developed as part of the study should enable significantly greater loadings to be achieved.

In addition to more careful tailoring of the NTO habit, incorporation of quantities of RDX or HMX into the formulation may be necessary to increase the performance of the formulation to that obtainable with Composition B. Although this would most probably adversely effect the IM properties of the formulation, newer so-called insensitive RDX being produced by SNPE may allow high performance insensitive general-purpose explosives to be developed that could be processed on existing

Australian plant with minimal alterations. (It must be noted, however, that the SNPE insensitive RDX has to date only shown improvements to reduced shock initiation properties in PBXs. This effect has been reported as being minimal in both melt-cast and pressed explosives).

It is recommended that a following series of work involve;

1. NTO recrystallisation studies to improve particle morphology and increase solids loading,
2. development of insensitive NTO/TNT compositions with increased NTO loadings and improved performance,
3. redesign of the stepped rate stick for critical diameter estimations using fewer and longer sections to minimise overdriving of the detonation front and an underestimation of $d_{crit}$.
4. examination of the effect of RDX incorporation on performance and vulnerability in ARX-4002 type formulations,
5. determination of the shock sensitivity of ARX-4002 along with the next series of developed compositions, and
6. preliminary formulation of the insensitive GP bombfill AFX-645.

# 7. Acknowledgments

The authors would like to thank Max Joyner for assistance in charge fabrication and machining, Richard Dexter for hazards assessment and Dave Harris, Dave Fraser and Rachael Campbell for conducting the firings and high-speed photography.

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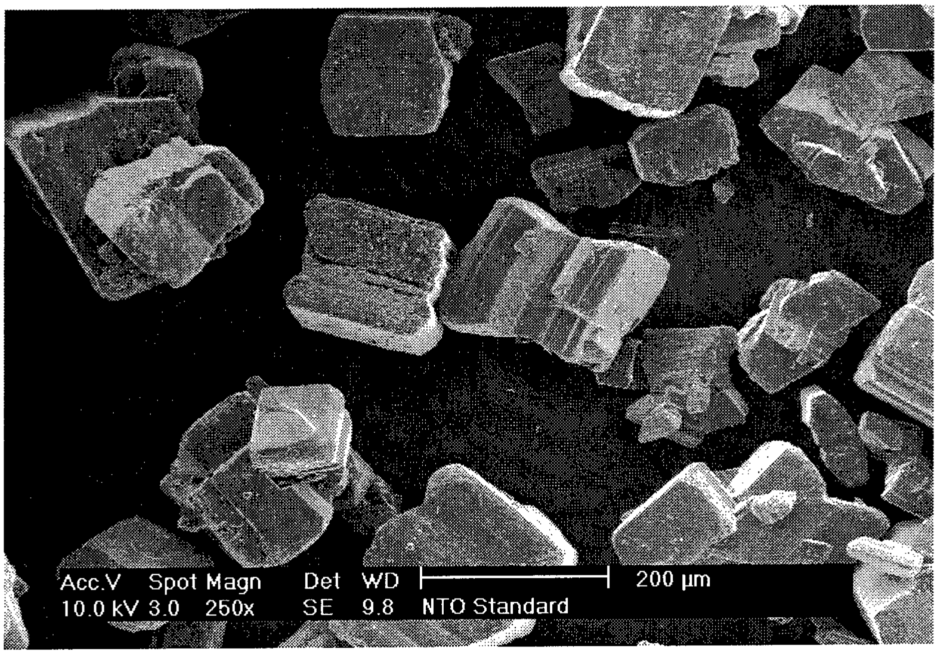


*Figure 1. SEM of medium grade NTO (200-400 μm) used in ARX-4002 formulation.*

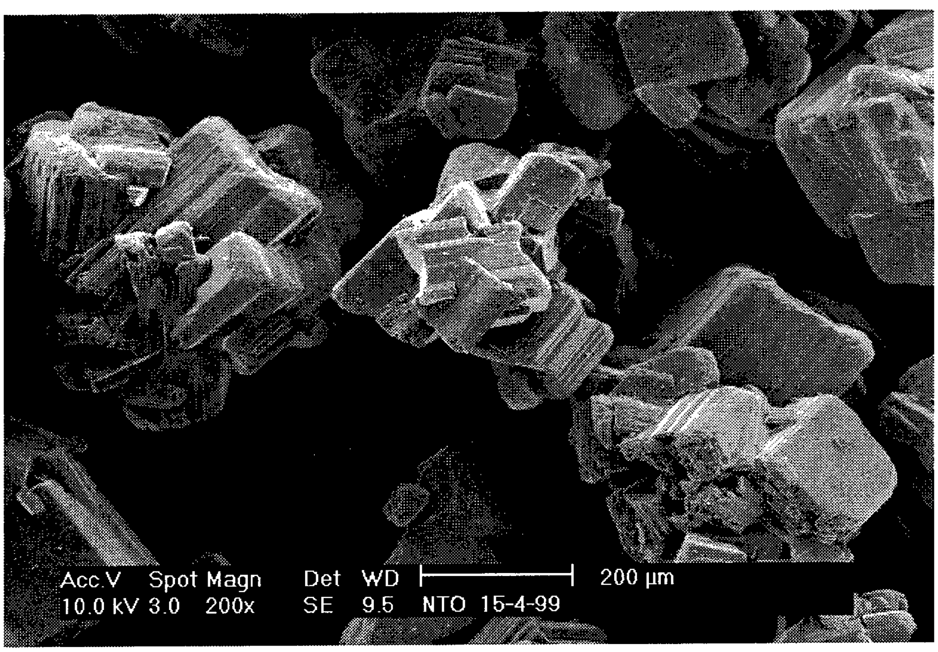


*Figure 2. SEM of medium grade NTO (200-400 μm) used in ARX-4002 formulation.*

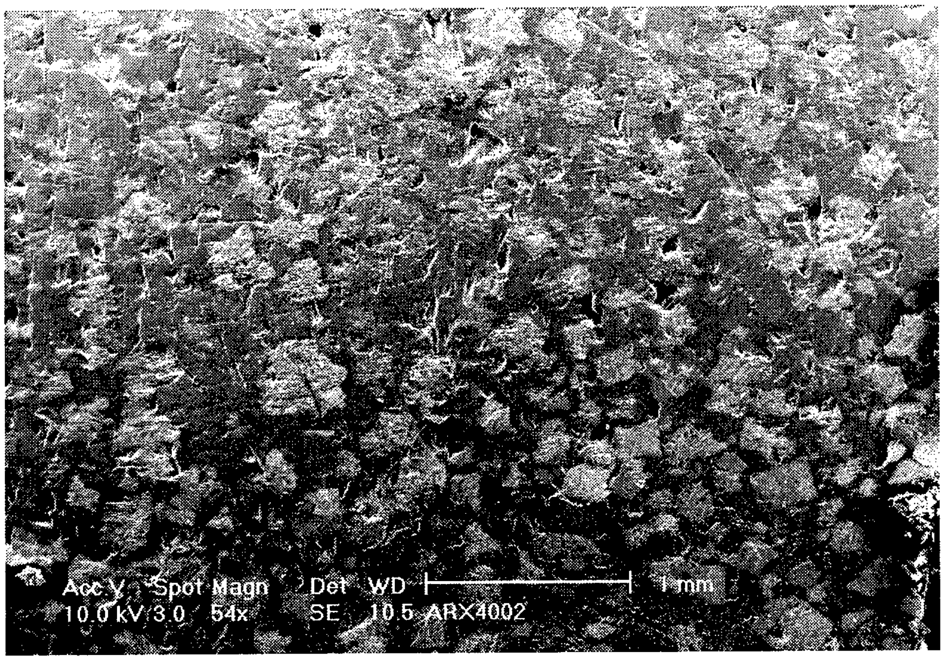


*Figure 3. SEM of polished ARX-4002 sample produced via Method 1. Numerous voids are present within the matrix due to occluded air.*

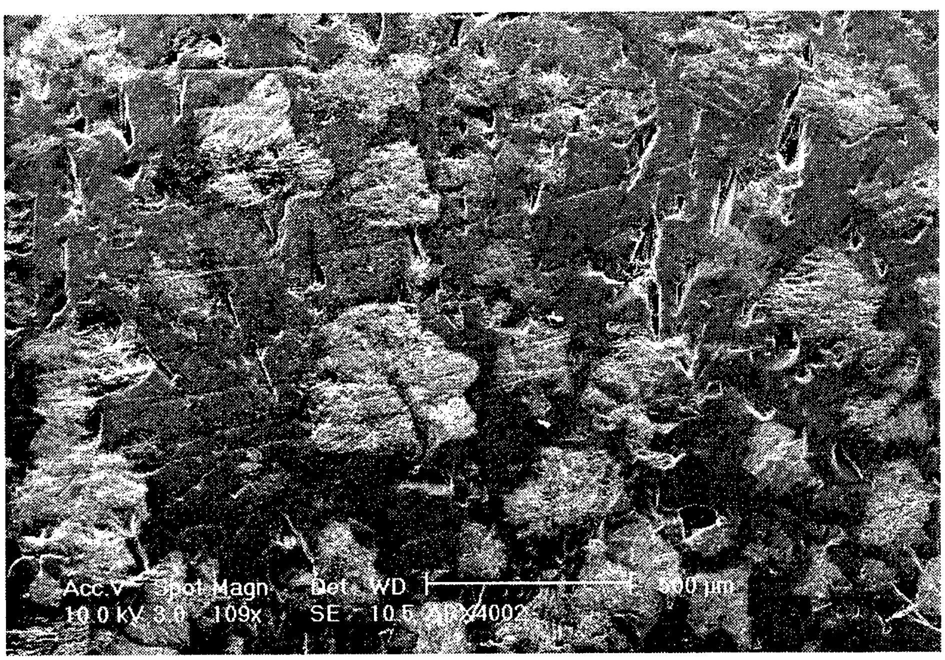


*Figure 4. SEM of polished ARX-4002 sample produced by Method 1.*

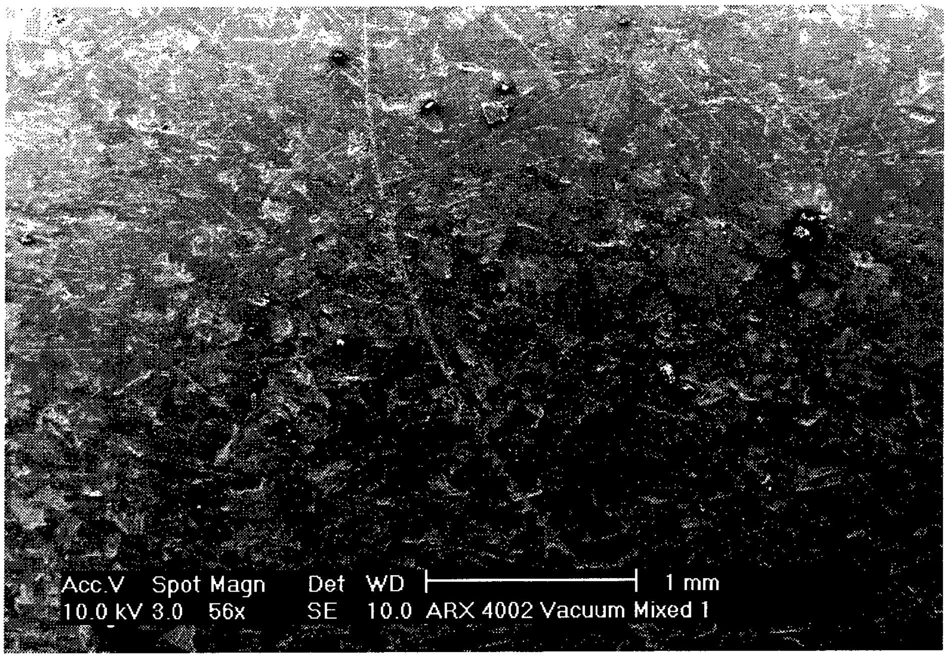


*Figure 5. ARX-4002 processed under vacuum* via *Method 2. NTO is well distributed in the matrix with no air voids present.*

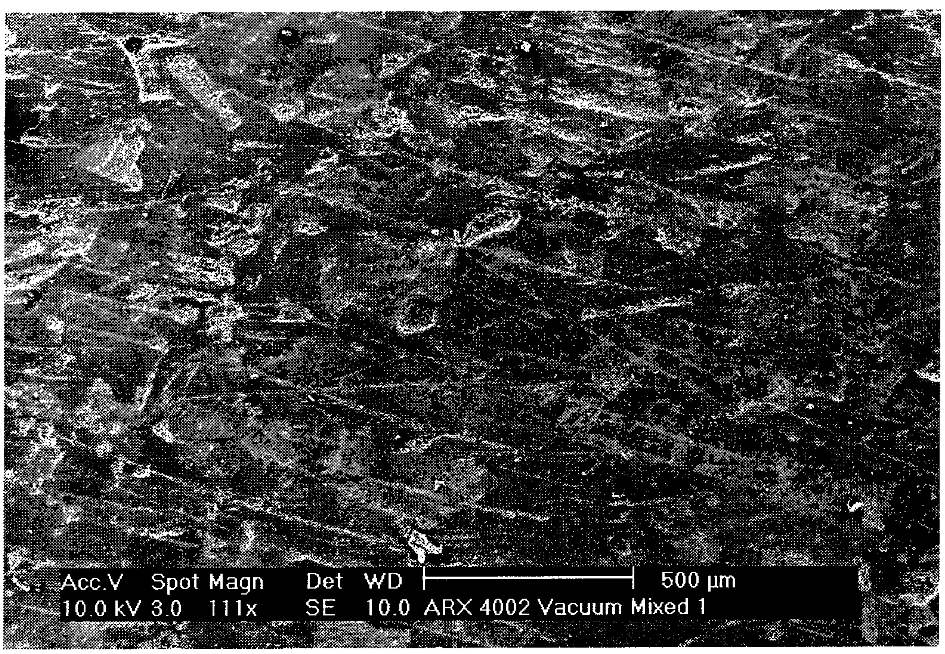


*Figure 6. ARX-4002 processed under vacuum* via *Method 2.*

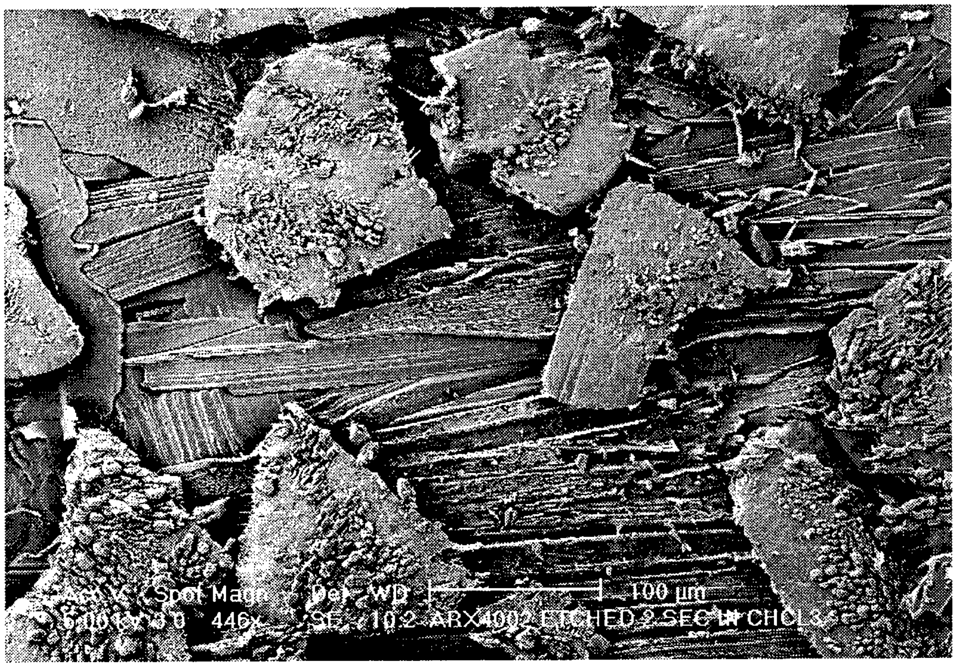


*Figure 7. ARX-4002 etched for 2 seconds in chloroform. Precipitated debris from the etching process has been deposited upon the NTO surface.*


*Figure 8. ARX-4002 etched for 5 seconds in chloroform. NTO crystals are standing proud from the TNT grain.*

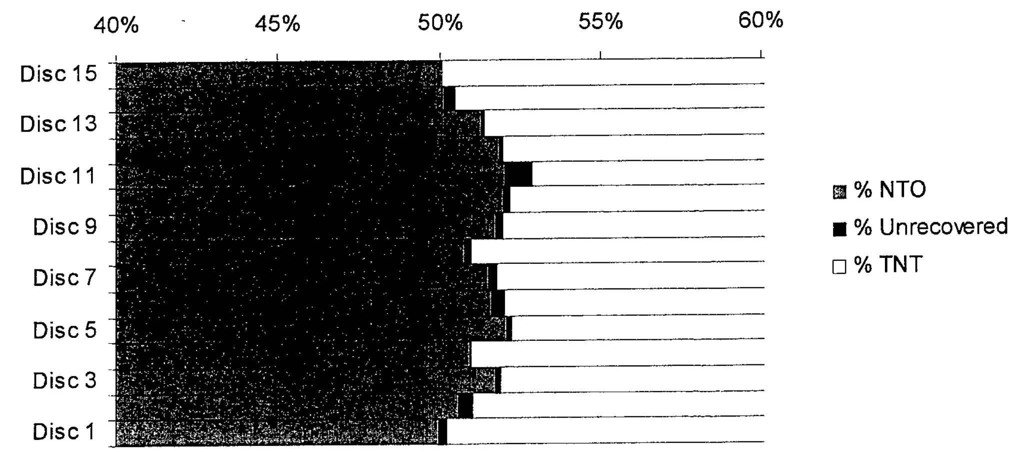


*Figure 9. NTO and TNT percentage composition of 25 mm diameter, sectioned ARX-4002 charge. Disk one at the base of the charge.*

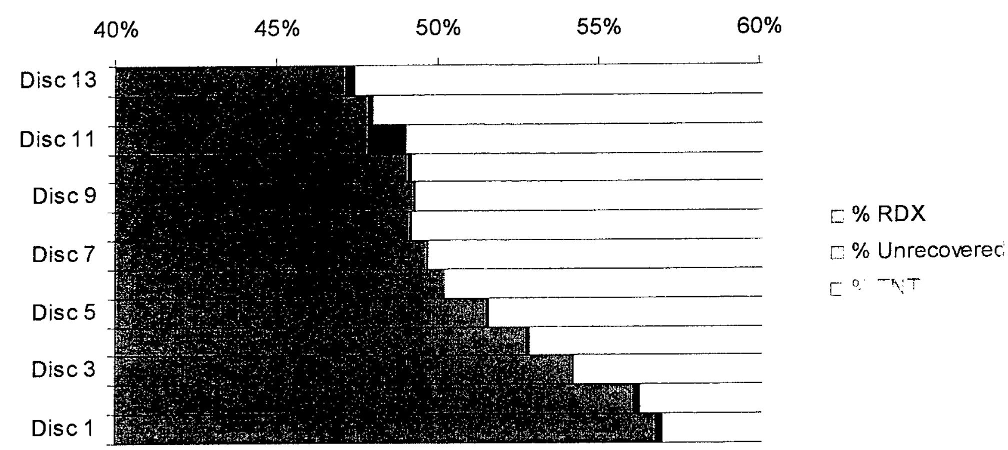


*Figure 10. RDX and TNT percentage composition of 25 mm diameter, sectioned [illegible] RDX/TNT reference formulation. Disk one at the base of the charge.*

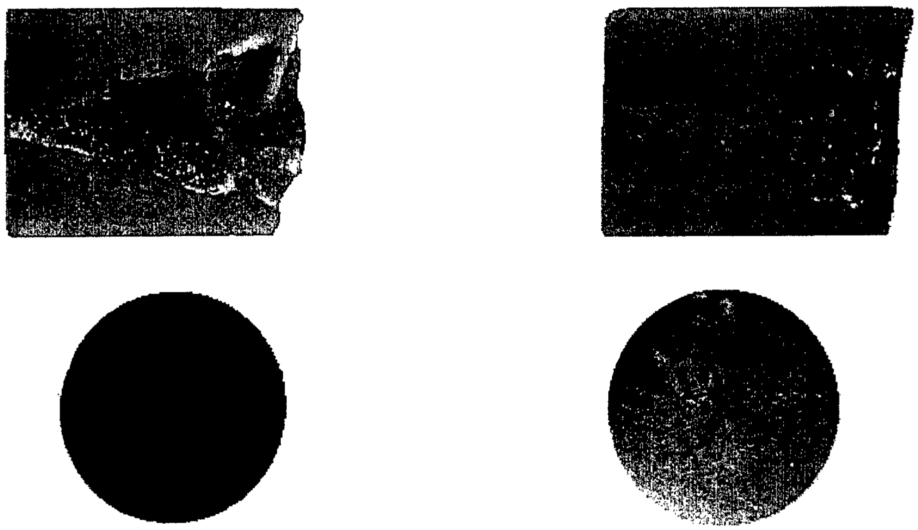

*Figure 11. Longitudinal section (upper) and cross-section (lower) images of the reference 50:50 RDX/TNT (left) and ARX-4002 (right) headers removed from the sedimentation sticks. ARX-4002 is less prone to coring than the RDX depleted header of the reference. (Both headers removed at the same level).*

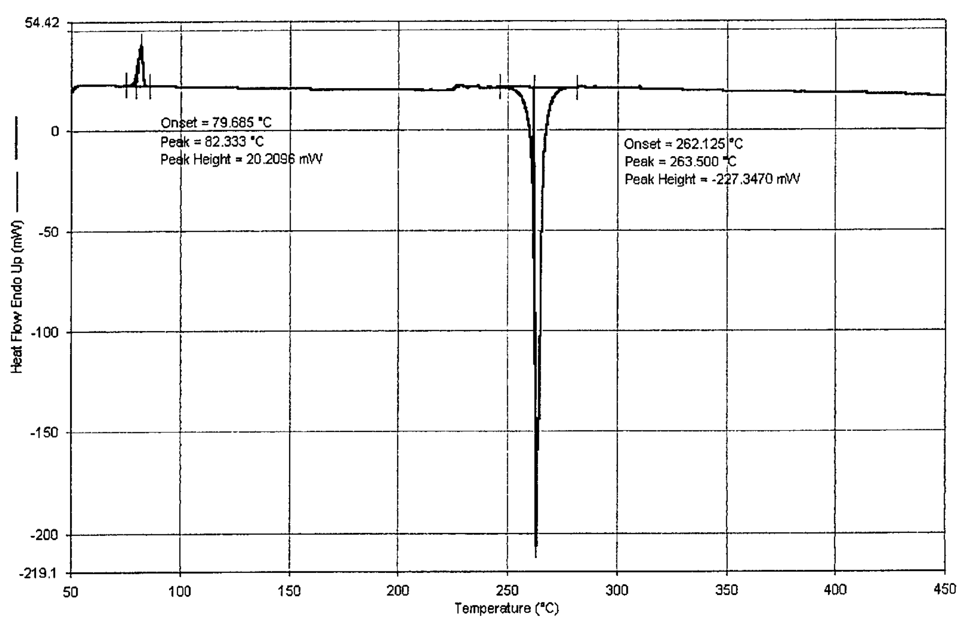


*Figure 12. DSC trace of ARX-4002.*

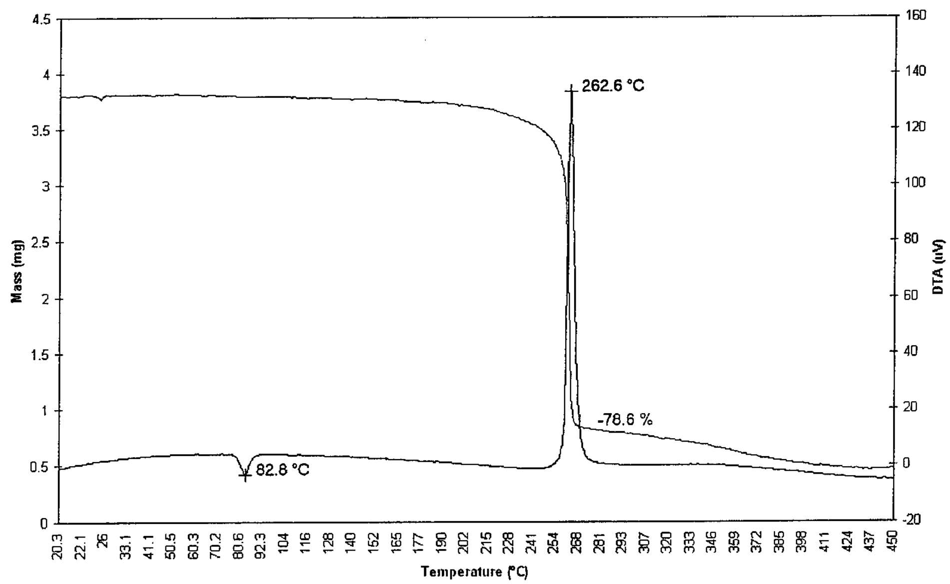


*Figure 13. TGA trace of ARX-4002.*

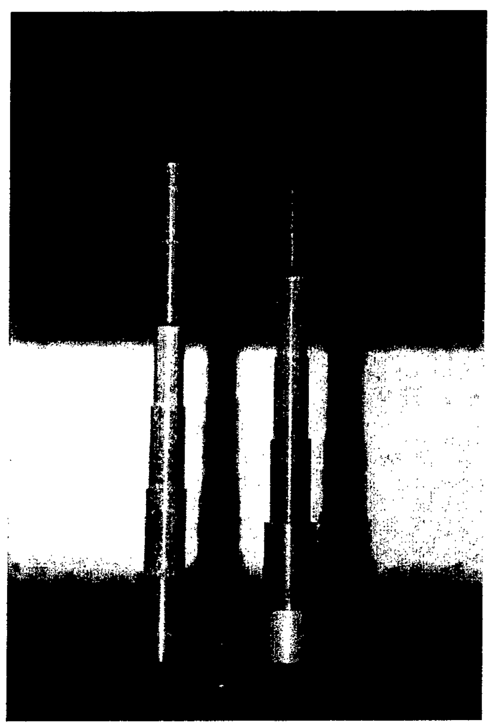

*Figure 14. Stepped charges for critical diameter estimations with (right) and without (left) attached 50:50 pentolite booster.*

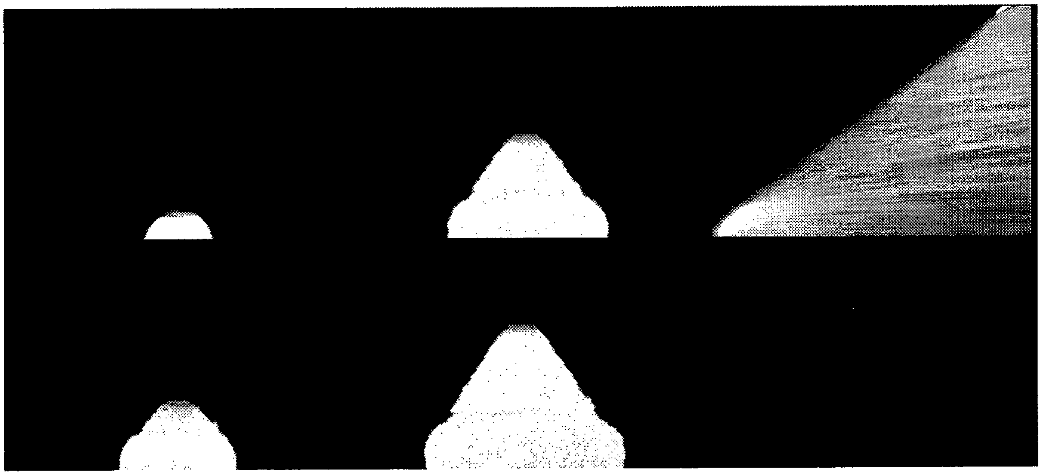

*Figure 15. Streak record of ARX-4002 critical diameter rate stick firing (25.0 mm diameter). Still frames at 8, 13, 18 and 23 μs from the firing pulse. Streak shows a steady state detonation of 7170 m/s.*

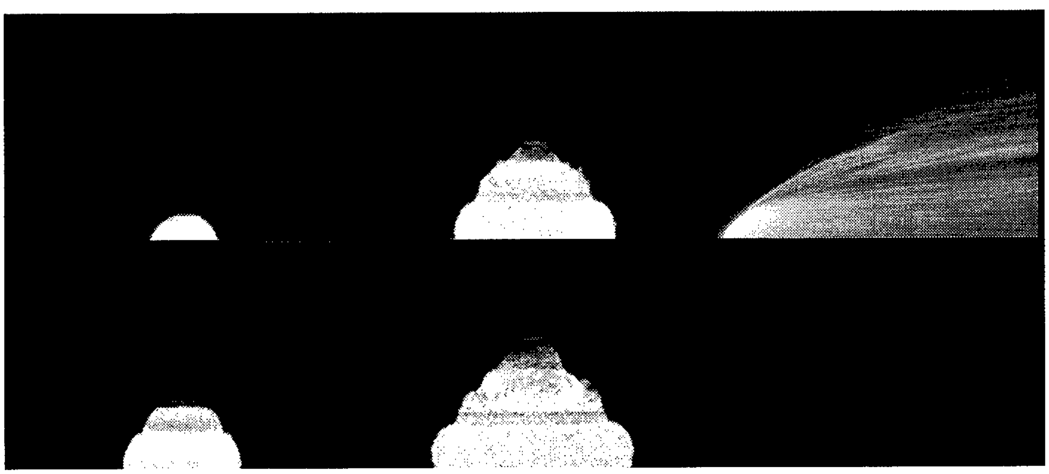

*Figure 16. Streak record of ARX-4002 critical diameter rate stick firing (22.5 mm diameter). Still and streak frames show detonation failure at this charge diameter.*

# Appendix A: Metallographic Polishing of NTO/TNT

## A.1. Primary Polishing

Initial polishing was performed using number 600 silicon carbide (automotive wet/dry) abrasive paper. Water was unable to be used for 'wetting' purposes due to the high solubility of NTO. Octane was found to be a suitable replacement due to the low solubility of both NTO and TNT in hydrocarbons and its relatively high boiling point (125-127°C) to minimise evaporation.

The carbide paper was wrapped around a flat plate of glass and placed in a shallow dish. The abrasive paper was wet with octane and the sample gently polished in a 'figure 8' motion until a flat surface is obtained. The abrasive paper must be washed with clean octane as necessary to prevent a build up of explosive particles for reasons of safety and to ensure the paper is not clogged.

## A.2. Intermediate Polishing

The intermediate polishing stage is to remove the rough, deeply scored surface left from primary polishing. The polishing process uses a finer, number 1200 silicon carbide paper wet with octane in the same manner as above. The sample is polished in a 'figure 8' motion taking additional care not to damage the surface.

## A.3. Finishing Polishing

Synthetic velvet cloth is stretched over a glass plate and octane applied to dampen the material. The sample is polished as above and the cloth washed regularly with clean octane. Attempts to use magnesium oxide/octane as a polishing medium resulted in a thick paste which stripped the colour from the velvet cloth. This technique was discontinued and octane-dampened velvet alone used.

## A.4. Etching

Etching of the polished samples was achieved by immersion into either chloroform or bromoform for a 2-5 second period. The sample is then removed and washed rapidly with octane to remove the solvent and dissolved TNT. A five second etching more fully reveal the TNT grain in ARX-4002.

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Matthew D. Cliff and Matthew W. Smith

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**Aeronautical and Maritime Research Laboratory**

Director
Chief of Weapons Systems Division
Research Leader: Dr J. S. Adams
Task Manager: Dr M. D. Cliff
Author: Mr M. W. Smith

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16. DELIBERATE ANNOUNCEMENT

No Limitations

17. CASUAL ANNOUNCEMENT Yes

18. DEFTEST DESCRIPTORS

Explosive compositions, fillers, NTO, trinitrotoluene.

19. ABSTRACT

Melt-castable NTO/TNT explosive formulations have been identified as possible candidates to replace RDX/TNT to meet Australian Insensitive Munitions obligations. Such formulations could be processed in existing ADI industrial facilities with minimal alterations to plant.

A baseline 50:50 NTO/TNT formulation, designated ARX-4002, has been developed. ARX-4002 can be processed using traditional melt-cast techniques and exhibits reduced sensitiveness in comparison to Composition B and H-6. The molten slurry is less prone to sedimentation problems, however, viscosities are increased and lower solid loadings possible than for Grade A RDX.

Velocity of detonation and detonation pressure were experimentally determined at 7370 m/s and 22.6 GPa respectively. Performance of ARX-4002 is thus less than for Composition B, however, the intrinsic sensitivity of ARX-4002 is expected to be greatly reduced as indicated by the large critical diameter (22.5-25.5 mm).